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(54) Title: LOW-EMISSION ELASTOMER FLOOR COVERING (54) Bezeichnung: EMISSIONSARMER ELASTOMER-BODENBELAG (57) Abstract <p>The present invention relates to a floor covering which causes substantially no emissions producing unpleasant smells and/or unhealthy emissions, and also does not change colour over a long period of time due to ageing. It also relates to a process for the production of said floor covering which contains an elastomer based on a polyolefine with a density of 0.918 g/cm³ acting as the polymer binding agent.</p> (57) Zusammenfassung <p>Die vorliegende Erfindung betrifft einen Bodenbelag, der im wesentlichen keine geruchsbelästigenden und/oder gesundheitsbeeinträchtigenden Emissionen verursacht, und darüberhinaus keine durch Alterung veränderbare Farbgebung über längeren Zeitraum zeigt, sowie ein Verfahren zu dessen Herstellung. Dieser Bodenbelag enthält ein Elastomer auf Basis eines Polyolefins mit einer Dichte 0,918 g/cc als polymeres Bindemittel.</p>		

Applicant: DLW Aktiengesellschaft
"Low-emission elastomer floor covering"
Our Ref: D 2572 - py / jh

Abstract

The present invention relates to a floor covering that causes essentially no unpleasant odors and/or health damaging emissions and which furthermore shows no signs of discoloration by aging for a lengthy time period, as well as a method for manufacturing the floor covering.



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Description

The present invention relates to a floor covering that causes essentially no unpleasant odors and/or health damaging emissions and furthermore will show no discoloration by aging for a lengthy time period, as well as a method for manufacturing the floor covering.

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Rubber-based elastomer coverings are one of the higher quality floor coverings because of their durability and high utility. Vulcanization and processing additives or agents have the tendency to emit chemical elements from the floor covering in their unchanged or chemically altered form.

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In general, rubbers of various types that may be vulcanized are used as polymeric binders for floor coverings. These rubbers are mainly SBR (styrene-butadiene rubber), NR (natural rubber), IR, IIR and NBR rubber whereby cross-linking is caused by cross-linking agents like sulfur, in combination with vulcanization additives. These vulcanization additives may be vulcanization accelerators like mercapto compounds, sulfenamides, thiuram, guanidine, dithiocarbamate, and amines; vulcanization decelerators like phthalic anhydride and N-cyclohexyl thiophthalimide, and preservatives like 2-mercapto benzimidazole; masticators like 2,2'-dibenzamido-diphenyl-disulfide; softeners or processing oils, strengthening resins like phenol-formaldehyde resin and vulcanization activators like zinc oxide. These cross-linking agents and additives are not totally converted during the vulcanization process. Thereby these additives or the by-products generated during the vulcanization process remain partially in the system or are emitted from this rubber system. This emission process may last for a long time period. This occurs in floor covering mainly when it is removed from packaging and is installed on the floor with an adhesive on a suitable backing. Temperatu-

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re, humidity and ventilation of an enclosed area further influence the process of emission.

In general, an unpleasant odor derives from the emitting vulcanization additives in the rubber system or from the by-products generated during the vulcanization process and this may also be health damaging in certain concentrations. It also has been shown that emitting substances from rubber-based floor coverings may stain white walls (plaster, wall paints etc.) under certain circumstances. Additionally, rubber-based elastomer floor coverings undergo an aging process that may be noticed as a disadvantage in the discoloring of design floor covering, for example by yellowing.

Therefore, the problem underlying the present invention is to provide a floor covering that is low in emission, resists the aging process, may be designed in variable colors, is aesthetic, is manufactured homogeneously and has elastomeric rubber-like qualities without the presence of vulcanized rubber.

This problem is solved by the embodiments characterized in the patent claims. In particular, a floor covering is provided that contains at least one elastomer based on a polyolefin with a density of $<0.918 \text{ g/cm}^3$ as a polymeric binder. The elastomer may be polyethylene with a very low density (PE-VLD) or a copolymer from ethylene with at least one additional olefin, like propene or butene. Preferably, a PE-VLD with a density of about $0.85\text{-}0.892 \text{ g/cm}^3$ is used, more preferred is a density of $0.86\text{-}0.87 \text{ g/cm}^3$.

The polyolefin used according to the invention, is cross-linked with a suitable cross-linking agent, an aromatic-free organic peroxide and with process enhancing additives like alkylenealkoxysilanes, trialkylencyanurate as well as dibutyltin dilaurate or mixtures thereof. For example, suitable cross-linking agents are as follows:



			Range weight %	Preferred range weight %
5				
10	peroxide	DHBP: 2,5 dimethyl-2,5di- (tert.-butylperoxy) hexan (Peroxidchemie Company)	0-4.0	0.1-1.2 (e.g. up to 0.4)
15	alkylenealkoxy- silanes	vinyltrimethoxyhydrosilicone or vinyltriethoxyhydrosilicone (Wacker Company)	0-4.0	0-1.0 (e.g. up to 0.4)
	trialkylene- cyanurate	trialkylencyanurate (Degussa Company)	0-3.0	0.6-1.0
20	catalyst	DBTL: dibutyltin dilaurate (Erbsloeh Company)	0-0.2	0-0.05

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The floor covering according to the present invention contains, for example, the above-defined thermoplastic polyolefin and elastomer, respectively, and its preferred ranges are as follows (weight percent):

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			Range [%]	Preferred value [%] example
35	Binder PE-VLD	DOW XU 5800052	25-75	53.5
		DOW DSK 8801	25-75	53.5
	Binder PE-VLD	Exxon Exact 4041	25-75	53.5
40		Exxon exact 4033	25-75	53.5

Furthermore, the floor covering according to the present invention may contain fillers or a mixture thereof. Examples of fillers and their preferred ranges are as follows (weight percent):

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	Fillers	Type example	Supplier	Range [%]	Preferred value [%] example
5	quartz powder	Sirkon SF 300	Quarzwerte GmbH	0-50	22.5
	kaoline	Nucap EDL 200	Lehmann & Voss & Co	0-20	8.5
10	talc	Alpha Talc CT 8/46	Alpha-Calcit Fuellstoffe GmbH KG	0-20	8.0
15	wood dust	Holzmuehle Westerkamp & Co	wood dust type 120	0-50	25
	wood dust	Holzmuehle Westerkamp & Co	wood dust type 70	0-50	25
20	dolomite	KL 30	Naintsch Mineralwerke Graz	0-40	20
25	aluminum trihydroxide	Martinal ON 313	Martinswerke GmbH	0-30	15
	precipitated silica	P 820	Degussa AG	0-40	20
30	heavy spar	Schwerspat TS	Sachtleben	0-50	22.5
	chalk	Omyacarb 6	Omya	0-50	22.5
35					

Furthermore, the following inorganic pigments may be contained, for example, in the floor covering according to the present invention depending on the color scheme and color matching, respectively (weight percent):

	Pigments	Supplier	Range [%]	Preferred value [%] (example)
40	Kronos 2200	Kronos Titan	0-8	3.5
45	Bayferrox 140	Bayer AG	0-3	"



Bayferrox 830	Bayer AG	0-3	"
Hostaprint A 2 H 31	Hoechst AG	0-3	"

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Optionally, the usual processing agents and process-enhancing additives, respectively, may be contained in the floor covering of the present invention. An example of such a processing agent is shown below (weight percent):

10	Slip additive	Type example	Supplier	Range [%]	Preferredvalue [%] (example)
	stearic acid	Barolub FTA	Barlocher GmbH	0.1-1.5	1.0

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In addition, antioxidants, UV stabilizers and such may be contained. Examples are as follows (weight percent):

20	Stabilizers	Type example	Supplier	Range [%]	Preferredvalue [%](example)
	antioxidant	Irganox 1010	Ciba Geigy AG	0-0.5	0.1
25	UV stabilizer	Chimassorb 944 FI	Ciba Geigy AG	0-0.5	0.1

30 By the elastomers used as a polymeric binder according to the present invention, the floor covering of the present invention has essentially no unpleasant odors and/or health damaging emissions in comparison to known floor coverings where SBR rubber is used as elastomer. Above all, the floor covering according to the present invention has a very high abrasion resistance in comparison to known rubber-based floor coverings. Surprisingly, the surface energy of the floor covering manufactured in such a manner may be increased by corona treatment so that the primer has better adhesion, which would result in better adhesive strength compared to rubber-based floor coverings. In addition, a lower discoloration (yellowing) during the life of the material has been found in the floor covering of the present invention. Thereby, a sufficiently flexible design and color scheme may be obtained in addition to a sufficiently aging resistance and

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low emission, while the desired elastomeric qualities still are maintained.

Another object of the present invention is a method for manufacturing the floor covering of the present invention, comprising the steps of providing a backing
5 in form of sheeting and applying the above defined elastomers on one surface of the backing.

All material used for backing in floor covering that has been used up to now can still be used, may it be natural and/or synthetic fabric, woven cloth, or textile
10 fabric. The following may be used, for example: Jute fabric, mixed fabrics made from natural fibers like cotton or viscose staple fiber, fiberglass cloth, fiberglass cloth coated with an adhesive agent, mixed fabrics made from synthetic fibers, fabrics made from mantle/core fibers like, for example a core of polyester and a mantle of polyamide.

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In addition, a new method for manufacturing a low-emission floor covering has been established, which comprises the following steps:

(1) Compounding a cross-linkable mass that contains the previously described
20 polyolefin and optionally at least one previously described process-enhancing additive and then preparing a sheeting by calender with subsequent milling to produce a grinding stock or direct granulating after extruder-compounding for the production of granules.

25 (2) Wetting of the grinding stock and granules (subsequently called "particles"), respectively, thus obtained with a solution that contains at least one aromatic-free organic peroxide used as a cross-linking agent and optionally white oil, whereby migration of peroxide into the grinding stock and granule-particles, respectively, is made possible.

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(3) Mixing the wetted grinding stock and granules, respectively, with a powder mix, which contains polyethylene (PE) powder and optionally soot and/or mixed with previously defined pigments and/or fillers and/or process-enhancing agents



and/or antioxidants and/or stabilizers and/or flame-retarding agents such as metalhydroxide, for example, whereby a free-flowing grinding stock and granules, respectively, are obtained mantled by the solution and the powder mix.

5 (4) Applying the thereby obtained free-flowing grinding stock and granules, respectively, on a backing or a suitable band and strip, respectively, where it is heated after pre-compressing by suitable means, for example by IR heater and/or hot air and the grinding stock and granules, respectively, are brought to a temperature of 160°C or 140°C, for example, whereby the peroxide has sufficient stability at this temperature, characterized by the half life period, for example: > 15 min or > 1 h.

10 (5) Press-molding the thereby pre-heated material in a double strip press, double band or steel band auma, under a suitable molding pressure of 1.2 to 2 bar/cm², for example, and at a temperature of e.g. 195 to 200°C, whereby the half life period for peroxide is decreased in such a manner that simultaneously a cross-linking of the material is initiated by the peroxide. For example, the peroxide DEHP has a half life period amounting to $t_{1/2}$ of 1 min at a temperature of 190°C.

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The texture or pattern of the cross-linked substance is uncovered after opening of the surface by grinding and/or splitting.

25 In another embodiment of the method according to the present invention, instead of the uncrosslinked mass used in step (1), there is used a partially cross-linked mass on the basis of the previously defined polyolefin, whereby a relief-texture of the flat product is obtained after press-molding in step (5). The partial cross-linking of the particles is caused during extruder compounding and before granulating, when granules are used, or during sheeting production and before milling, when the grinding stock is used. The partially cross-linked particles are deformed reversibly during press-molding and a raised or embossed texture is obtained by the restoring force after pressure release. The degree of cross-linking of the partially cross-linked substance may be adjusted by the

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quantity of peroxide.

The heating described in step (4) further enhances migration of the cross-linking agent into the grinding stock and granule-particles, respectively.

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Another embodiment of the method according to the present invention is shown in step (1) where a cross-linkable mass, based on the previously defined polyolefin, is compounded with at least one cross-linking agent and optionally with a previously defined additive, whereby partially cross-linked granules may be
10 obtained by controlling the time the substance is in the extruder (residence time) and the corresponding screw shape and the length of the extruder and by controlling the heating of partially cross-linked granules. According to this embodiment, the same possible patterns may be produced as with PVC technology. That means that a pre-determined texture may be obtained, for example.
15 In particular, mechanical characteristics of elastomer floor coverings may be combined with the optical/texture characteristics of PVC floor coverings with this embodiment of the present invention.

In a further embodiment of the method according to the present invention, the
20 mass in step (1) may be compounded additionally with a chemical foaming agent like sulfohydrazides or azodicarbonamides or combinations thereof, for example Lovopor 1417 (Lehmann & Voss Co.) or Tracel DB 145 (Tramaco GmbH). After cross-linking under pressure as given in step (5), foaming of the substance is obtained after pressure release. This embodiment may be utilized especially for
25 floor coverings with foamed backings.

The advantages of the method of the present invention are especially the use of a powder mix containing polyethylene to make the wetted grinding stock and granules, respectively, flow freely, the stability of the peroxide compound as
30 cross-linking agent up to the heating carried out in step (4) of the method of the present invention. By the use of an uncrosslinked or partially cross-linked mass, which means a cross-linkable mass without peroxide or with a very low amount of peroxide, the process may be controlled through the migration of the peroxide



used in the solution for mantling of the grinding stock and granule-particles, respectively.

The figures illustrate the following:

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Figure 1 is a schematic illustration of a mechanism and apparatus, respectively, for the production of the floor covering of the present invention (see example 1).

10

Figure 2 is a schematic illustration of a preferred embodiment (in the direction of the arrow) for the production of plastic sheeting according to the present invention (see example 2).

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Figure 3 is a schematic illustration of a preferred embodiment for the production of the floor covering of the present invention (see example 3).

Figure 4 is another schematic illustration of a preferred embodiment for the production of the floor covering of the present invention (see example 4).

20

The present invention is explained in more detail by the following examples:

Example 1

A double-screw extruder Type ZE 40 made by Berstorff with $L/D = 40$ at $D = 40$, functions as a preparation aggregate, for example. The gravimetric measured compound mixture (PE-VLD, fillers, process enhancing agents, pigments) is plasticized and homogenized within a lengthy intake area (10D) by using suitable transporting and kneading elements. By means of a precision metering pump, the mixture for grafting consisting of vinyltrimethoxysiloxane (VTMOS), organic peroxide (DHBP) as well as dibutyltin dilaurate (DBTL) as a catalyst is fed over a cooled metering lance into the compound mass that is maintained at a temperature of 120°C . In this mixing phase the peroxide does still not decompose because of the low temperature. By subsequent increasing of the temperature in the following extruder elements, peroxide decomposes and initiates grafting of



VTMO at PE-VLD by radical reaction and also initiates cross-linking of PE-VLD with one another. Thereby, PE-VLD loses its thermoplastic character that is necessary for molding. A vacuum de-gassing device removes volatile reaction products (ethanol, methanol, n-isobutanol) in order not to emit later into the ambient atmosphere. The cross-linked PE-VLD is again degraded in the last kneading zone to make the substance thermoplastic once more. The mass is either granulated according to the pattern design - various granule colors are statically press-molded as a granule mixture - or it is made into sheeting by feeding it through a wide slotted nozzle onto an adjacent smoothing roll - whereby subsequently colored designed granules are pressed into the still heated sheeting by a backing roll. The floor covering is then smoothened in a continuous press. The cross-linking is caused by aqueous initiation and condensation reaction of methylsiloxane or silanol in the commonly known manner.

15 Example 2

A Banbury mixer (plunger-type mixer) or a double-screw mixer (e.g. ZE 40, Berstorff) functions here as a preparation aggregate by choice. In this example, a cold pre-mixed mixture is directly fed into the mixer together with peroxide (without VTMOs, DBTL). The compounding is performed within 4-7 minutes and preferably under a temperature of 140°C (135°). This substance is subsequently fed through a wide slotted nozzle and a smoothing roll or a press at a temperature of 120-130°C and then processed into sheeting. This sheeting can now possibly be made into granules, which then may be mixed and press-molded with other designed granules or the sheeting may be sprinkled with differently designed granules and subsequent continuously fed into a double-band press at a pressure of 0.1 kg/cm² to 5 kg/cm², but preferably in the range of 1.5 to 2.5 kg/cm². The temperature is raised above the decomposition temperature of the peroxide (190-210°C) during press-molding and cross-linking of the PE-VLD is initiated within 1-3 minutes. The product is then cooled down to approximately 80-110°C in a cooling area and is tempered at a temperature of 80-90°C.



Example 3

A Banbury mixer (plunger-type mixer, internal mixer) functions as a preparation aggregate whereby the pre-mixed mixture is here also fed directly into the mixer and is compounded there preferably at a temperature of 135°C within 4-7 minutes. The mass is subsequently processed into sheeting by means of a press at a temperature of 120-130°C. Patterns may be produced whereby the already cross-linked (which means duroplastic or elastomeric) color contrasting granules are either added into the Banbury mixer in the final stages of the kneading process or are sprinkled into the to-be-kneaded built-up substance in the press. The first possibility is more advantageous since there is a more even distribution of granules. Subsequently, the sheeting surface is uncovered, depending on the degree of smearing of the designed texture, by grinding or continuous splitting (similar to the leather processing technology). The product is later embossed and cross-linked in a continuous press at a pressure of 0.1 kg/cm² to 5 kg/cm², but preferably 1.5 to 2.5 kg/cm². The temperature is brought above the decomposition temperature of the peroxide (190-210°C) during press-molding and cross-linking of the PE-VLD is initiated within 1-3 minutes. The product is then cooled down to a temperature of approximately 80-110° in a cooling area and is then tempered at a temperature of 80-90°C.

Example 4

In this example, the uncrosslinked granules are either produced by the Banbury mixer/ press granulating technology, whereby the final mixed mixture is compounded at a temperature of 135°C, is press-molded at 120-130°C, and then cold granulated (preferably 8-10 mm). Subsequently, the granules are mantled with a paste in a mixing drum consisting of the following substances:



	Substance	Type/Make	Supplier	Weight. %
	white oil	Dealen CP 31 N	DEA	15-50
5	or			
	conductive soot	Sicoplast V 00 6053	Sigle	8-50
	or			
10	multi-colored pigments as described	Bayferrox 140 Bayferrox 530 Hostaprint A 2 R 31 Kronos 2200 Sicoplast V 00-8920	Bayer Bayer Hoechst Kronos Titan BASF	5-50
15				
	dispersant	glycerin monostearate	Hoechst	0-1
20	extender	Omyacarb 6	Omya	0-30

The portion of paste per kilogram of granules amounts to 5-25 weight percent, preferably 8.5 weight percent. The mantled granules are now press-molded into sheeting in a continuous press, calibrated to a certain thickness and simultaneously cross-linked at a temperature of 200°C, if the surface of the floor covering is not to be embossed. The uncovering of the actual floor covering pattern is performed by a choice of splitting technology or grinding of the multi-colored smeared outer skin.

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A floor covering may be produced that has an electric conduction resistance of less than 10^{-3} ohm (measured according to DIN 54346) by applying with a pug mill an electrically conductive soot paste to the functional granules and by subsequent press-molding into sheeting.

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It has been proven in tests that press-molding should be conducted without cross-linking should the surface be embossed. Embossing is performed after uncovering of the surface, whereby it is simultaneously cross-linked during continuous contact at 190-210°C and at a pressure of 0.1 kg/cm² to 5 kg/cm², but preferably 1.5 to 2.5 kg/cm². The temperature is brought above the decom-



position temperature of the peroxide (190-210°) during the press-molding process and cross-linking of the PE-VLD is initiated within 1-3 minutes. The product is then cooled down to a temperature of approximately 80-110° in a cooling zone and is tempered at a temperature of 80-90°C. A special optical effect may be obtained by the application of black or multi-colored pigments. Surprisingly, it has been shown that oil in the mantle may migrate into the outer area of the granules because of the outstanding oil-compatibility of PE-VLD and thereby pigments are more likely to be present in the outer areas.

Evaluation of examples 1, 3 and 4

Example 1

	Test H182 Procedure 1	rubber-based floor covering Product 1	rubber-based floor covering Product 2
Exact 4033	53.5		
Omyacarb 6	23.1		
Sirkon SF 300	21.4		
Silan SF 300	0.5		
DHBP	0.5		
DBTL	0.01		
Barolub FTA	1.0		
Parameters			
Zone 1 [°C]	135		
Zone 1	135		
Zone 1	135		
Zone 1	135		
Zone 1	190		
Zone 1	210		
Zone 1	210		
Zone 1	180		
extruder speed	220		
[rotations/min]			
granulator matrix [mm]	9x9		
double-band press			
press conditions,	2,2		
pressure			
[kg/cm ²]			



	temperature			
	Zone 1 [°C]	180		
	Zone 2	200		
	Zone 3	120		
5	floor covering thickness			
	calibration [mm]	4.1		
	splitting of sheeting			
10	into two parts [mm]	2.0		
	surface energy [mN/m]			
		28.8		
	surface energy after			
15	corona [mN/m]	56.2		
	primer (g/m ⁴)	20.0		
	wet application	Intrafol-D 1 151/2 (Fuller Company)		
20	technical measurements			
	peeling values			
	DIN 16860	3.4	1.3	1.4
	[N/mm]			
25	adhesive Supra Strong (Wulf Company)			
	embossing			
	characteristics EN 433	90.8	83.4	83.2
30	recovery [%] 150 min.			
	Shore A hardness	85	90	89
	Shore D hardness	31	41	44
35	density [g/cm ³]	1.259	1.529	1.666
	abrasion characteristics			
	DIN 53516,			
	ISO 4649, prEN	94.4	163.8	259.9
	6 [mm ²]			
40	tension testDIN 53504			
	tension force			
	F-max [N/mm ²]	9.1	7.6	7.8
45	tensile force F-breaking	9.1	7.5	7.7
	max. tension force			
	stretching[%]	803	75	28
	further ripping resistance			
		36.7	27.6	21.4



	lengthwise	34.1	18.9	24.3
	DIN 53515 [N/mm]			
	crosswise			
5	burning characteristics DIN 4102 part 14 "B1"			
	burning distance [cm]	18	25	27
10	smoke density integral of %(transmission) x min.	32	675	552
	humidity			
	volume increase	0.038	0.077	0.083
15	humidity absorption [%]	0.14	0.56	0.92
	total emission-VOC according to Flec-method GC MS [$\mu\text{g}/(\text{m}^2 \times \text{h})$] * (1			
20		101	6744	11465
25	Example 3			
		Test H 246	rubber-based floor covering	
		Procedure 3	Product 1	
30	DSH 8501	53.5		
	DSH 58.000.52			
	Omyacarb 6	23.1		
	Sirkon SF 300	21.4		
35	P 820			
	Martinal-OL 313			
	Silan XL 10	0.5		
	DHBP	0.5		
	DBTL	0.01		
40	Baerolub FTA	1.0		
	Parameters			
	Banbury mixing time [min]	4.5		
	Banbury temperature [°C]	120		
45	Mass temp after Banbury [°C]	135		
	parts of decorative colors [%]	8		



5	grain size of granules manufactured in this procedure [mm]	2-3.5	
	measured into mixer		
10	press temperature	130°C	
	grinding of sheeting [m/min]	4	
	press smoothing temp pressure [kg/cm ²]	135°C 1.1	
	embossing	embossing roll in press	
15	cross-linking in double-strip press	200°C	
	press pressure [kg/cm ²]	1.1	
	floor covering thickness calibration [mm]	2.0	
20	surface energy [mN/m]	26.1	
	surface energy after corona [mN/m]	52.3	
	primer [g/l]	20.0	
25	wet application	Intrafol D 1151/2 (Fuller Company)	
	technical measurements		
30	peeling values DIN 16860 [N/mm]	3.3	1.3
	adhesive Supra Strong (Wulff Company)		
35	embossing characteristics		
	recovery [%] 150 min. EN 433	91.3	83.4
40	Shore A hardness	86	90
	Shore D hardness	31	41
	density [g/cm ³]	1.282	1.529
45	abrasion characteristics		
	DIN 53516, ISO 4649, prEN 6 [mm ²]	79.8	163.8



tension test		
5	tension force DIN 53504	
	F-max [N/mm ²]	9.3
	tensile force, F-breaking [N/mm]	7.6
10		
	max. tension force stretching [%]	9.0
		7.5
15	further ripping resistance lengthwise	
	DIN 53515 [N/mm]	36.5
	crosswise	27.6
		34.1
		18.9
20	burning characteristics DIN 4102 part 14 "B1"	
	burning distance	21
	smoke density	25
	humidity	675
25	volume increase [%]	0.042
	humidity absorption [%]	0.077
	total emission-VOC after Flec-method GC-MS [$\mu\text{g}/(\text{m}^2 \times \text{h})$] *(1	0.15
30		0.56
		155
		6744

Example 4

35	Test H289 Procedure 4		rubber-based floor covering Product 2
40	Parameters		
	DSH 8501		
	DSH 58,000.52	49.5	
	Omyacarb 6		
45	Sirkon SF 300	16.8	
	P820	19.3	
	Martinal OL 313	12.6	
	Silan XL 10		
	DHBP	0.8	



	DBTL	
	Barolub FTA	1.0
	Parameters	
5	Zone 1 [°C]	130
	Zone 2	130
	Zone 3	
	Zone 4	
	Zone 5	
10	Zone 6	
	Zone 1	
	Zone 1	
<hr/>		
	extruder speed	
	[rotations/min]	
15	granulator matrix [mm]	9x9
	granule mantled	
	with paste	
	sprinkling on double-strip	
20	press [mm]	6-6.5
	double-band press	
<hr/>		
	press conditions,	
	pressure	2.2
25	[kg/cm ²]	
<hr/>		
	floor covering thickness	
30	calibration [mm]	4.1
<hr/>		
	splitting of sheeting	
	into two parts [mm]	2.0
<hr/>		
35	embossing	
<hr/>		
	cross-linking in double-strip	
	press	
	press pressure [kg/cm ²]	
40	temperature	
	Zone 1 [°C]	180
	Zone 2	200
	Zone 3	120
45		



floor covering thickness
calibration [mm]

5	splitting of sheeting into two parts [mm]	2.0	
	surface energy [mN/m]	25.9	
10	surface energy after corona [mN/m]	50.1	
	primer (g/m ⁴)	20.0	
	wet application	Intrafol D 1151/2 (Fuller Company)	
15	technical measurements		
	peeling values		
20	DIN 16860	5.1	1.4
	[N/mm]		
	adhesive Supra Strong (Wulf Company)		
25	embossing characteristics		
	recovery		
	EN 433 90.8		
	[%] 150 min.	93.1	83.2
30	Shore A hardness	89	89
	Shore D hardness	46	44
	density [g/cm ³]	1.354	1.666
	abrasion characteristics		
	DIN 53516,		
35	ISO 4649, prEN	67.3	259.9
	6 [mm ²]		
	tension test		
	tension force		
40	F-max [N/mm ²]	9.6	7.8
	DIN 53504		
	tensile force F-breaking	9.4	7.7
	max. tension force		
	stretching[%]	356	25
45	further ripping resistance	40.2	21.4
	lengthwise	38.6	24.3
	DIN 53515 [N/mm]		
	crosswise		



 burning characteristics DIN 4102
 part 14 "B1"

5	burning distance [cm]	20	27
	smoke density	33	552

 humidity

10	volume increase [%]	0.023	0.083
	humidity absorption [%]	0.14	0.92

 total emission-VOC
 according to Flec-method GC
 MS [$\mu\text{g}/(\text{m}^2 \times \text{h})$] *(1

15		91	11465
----	--	----	-------

 *(1: Flec-method

20 measuring parameters: FLEC - ATD GC/MSD

 flow Flec: 100 ml/min desorption time: 5 min FID separation column: HP-1
 flow Tenax: 2 + 40ml/m desorption temp: 200°C MSD sep. column: HP-5
 MS

25	rel. flow %	50%	cooling drop temp.	-20°C	start temp:	50°C
	Temp:	23°C	cooling drop temp	300 °C	end temp:	280°C
	Tenax:	100mg	heating completion time:	2 min	heating rate	5°/min
	Time	24h	transfer temp:	275°C	low mass:	45
	blind test	1h	split	10 ml/min	high mass:	550

Example 5

Granules are placed into a Banbury mixer or a double-screw mixer for compounding whereby the granules contain 35 parts by weight of DOW XU 58000.52, 20 parts by weight of Sillitin N85 (Hoffmann Company, FRG), 0.5 parts by weight of stearic acid ("ETA") and 0.3 parts by weight of triallylcyanurate ("TAC"). Subsequently, the obtained granules are wetted with a solution that contains, for example, 0.5-3 or 2 parts by weight of Dealen CP 31.N (DEA) and 1 part by weight of DHBP. These wetted granules are mixed with a powder mix that contains 20 parts by weight of DOW NG 2431.10 E (DOW), 5 parts by weight of Kronos 2200 (Kronos Company), 10 parts by weight of Martinal ON 310 (Martinswerk Company) and 2 parts by weight of multi-colored pigments. This is done to obtain the necessary flow of particles for further processing.



These thereby produced unmantled granules are sprinkled on a suitable backing and are heated to a temperature of 160°C by a IR heater and/or hot air after being pre-compressed. The pre-heated material is press-molded at a temperature of 195 to 200°C under a pressure of 1.2-2 bar/cm² and is simultaneously cross-linked. The uncovering of the floor covering pattern may be performed by splitting technology or by grinding of the outer skin.

The floor covering obtained according to this example has a very low emission of volatile elements in addition to the outstanding qualities of the material.

10 Example 6

The processing is the same as in example 5, with the exception that further additional parts of a partially cross-linked mass containing 35 parts by weight of DOW X2 58000.52, 20 parts by weight of Sillitin N85, 0.5 parts by weight of FTA, 0.4 parts by weight of DEHP and 0.3 parts by weight of TAC is used for the preparation of the initial granules. A relief texture of the flat product may be obtained by the use of the partially cross-linked substance, which is essentially completely cross-linked during press-molding by migration of the peroxide into the granule particles. The thereby produced floor covering is also less slippery when wet in addition to the special optical effect.

Example 7

25 Affinity EG B200 is compounded with Luparco 231-SRA-40 in the extruder at a temperature of 190°C, whereby partially cross-linked granules are obtained. The further processing steps are the same as in example 6.

30 From the results it becomes apparent that the innovative floor coverings have a considerably lower emission of volatile substances than currently known state-of-the-art rubber-based floor coverings. Furthermore, the innovative floor coverings have an outstanding resistance to chemicals, are aging resistant and are resistant to abrasion.



Claims

1. A method for manufacturing a floor covering, comprising the following steps:

- wetting of particles with a solution that contains at least one aromatic-free organic peroxide and white oil, wherein said particles contain uncrosslinked or partially cross-linked elastomer, based on a polyolefin with a density of <0.918 g/cm as a polymeric binder, in form of a grinding stock or granules,
- mixing of said wetted particles with a powder mix containing a polyethylene (PE) powder for the production of free-flowing particles,
- placing said free-flowing particles on a strip, and after pre-compressing of said particles, heating the pre-compressed particles to a temperature at which the peroxide has a sufficiently lengthy stability, and
- press-molding the pre-heated particles in a suitable device at a temperature whereby the half life period of said peroxide is decreased in such a manner that cross-linking initiated by said peroxide occurs simultaneously to obtain a flat product.

2. The method according to claim 1, wherein said elastomer is polyethylene or a copolymer made from ethylene with at least one other olefin.

3. The method according to claim 1 or 2, wherein said polyolefin has a density of 0.86 to 0.87 g/cm³.

4. The method according to one of the claims 1 through 3, wherein the particles are compounded with at least one cross-linking agent and a process enhancing additive in the initial step .

5. The method according to claim 4, wherein said process enhancing additive



is selected from alkoxysilanes, alkylenealkoxysilanes, cyanuric acid derivatives or mixtures thereof.

5 6. The method according to one of the claims 1 through 5, wherein the powder mix contains additional fillers and/or pigments as well as optionally process enhancing agents, antioxidants and UV stabilizers.

10 7. The method according to one of the claims 1 through 6, further comprising the step of designing the floor covering in various colors.

8. The method according to one of the claims 1 through 7, wherein the floor covering is of homogeneous construction.

15 9. The method according to one of the claims 1 through 7, comprising the steps of providing a backing in the form of a sheeting and applying an elastomer as defined in anyone of the preceding claims on one side of said backing.

Dated this 17th day of May, 2000
DLW AKTIENGESELLSCHAFT
by their Attorneys
AHEARNS



I/4

Fig. 1

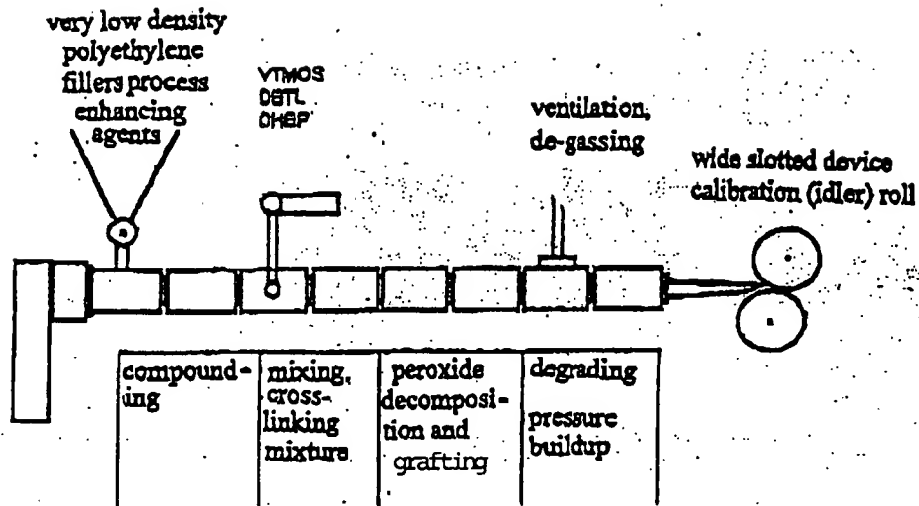


Fig. 2

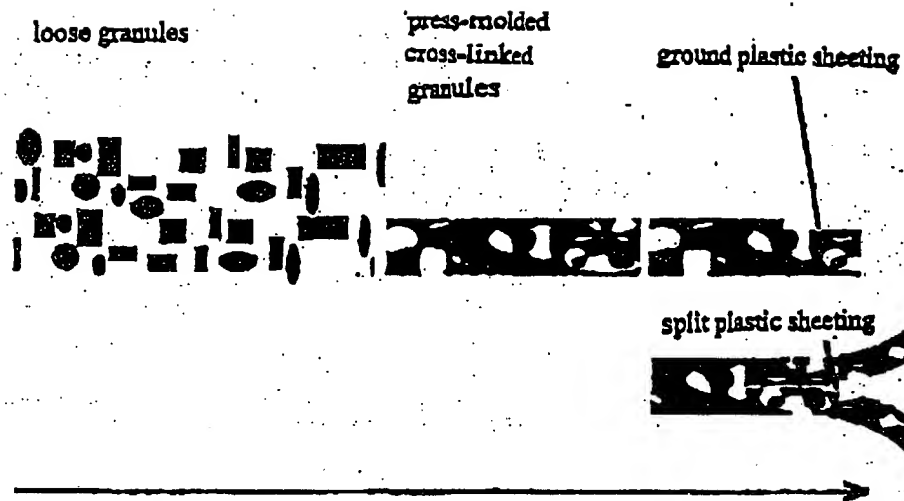


Fig. 3

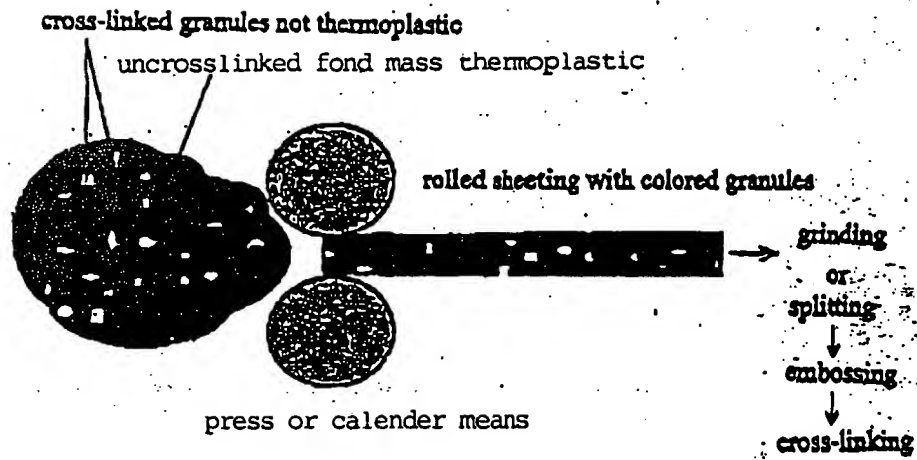


Fig. 4

